WORK INSTRUCTION
pH Determination of Mainstream Smoke in an Aqueous Solution

Objective:

To measure the pH of mainstream cigarette smoke in an aqueous solution through the analysis of a combined sample of smoke collected in water (using glass impingers) and TPM (collected on a Cambridge filter pad). Cigarettes are smoked in accordance with Federal Trade Commission (FTC) guidelines using a Filtrona 20-port smoking machine. All equilibration, smoking, and pH measurements are conducted under FTC conditions, 75 °F ± 2 °F and 60 % ± 2 % relative humidity. Four (4) replicates are measured per determination.

Apparatus & Instrumentation:

1. Accumet pH/ion meter, model 25, Cat. #13-635-25, Fisher Scientific.
2. Accumet glass body combination electrode, Ag/AgCl internal reference element, Cat. #13-620-285, Fisher Scientific.
3. Glass Impingers, (smoke collection traps), Research Glass.
4. Magnetic stir plate, Cat.# 27 25 001-7, Brinkmann.
5. Filtrona Model 400 Smoking Machine.
6. Plastic Microswitch Handlebars.
7. Cambridge Filter Pads, 44mm, Filtrona.
8. Borgwaldt Lighter, KC Automation.
9. Borgwaldt Soap Bubble Meter, KC Automation.
10. Schiltknecht Air Flow Meter, KC Automation.

Chemicals:

1. Buffer Solutions, pH 4.0 Cat.# SB101-500, pH 7.0 Cat.# SB107-500, and pH 10.0 Cat.# SB115-500, Fisher Scientific.
2. Hydrogen Peroxide 3%, Cat.# H324-500, Fisher Scientific.
3. 4M KCl saturated with silver chloride electrode filling solution, Fisher Scientific.
4. Degassed Deionized Water, 18 megaohms-cm, (water is supplied from a Milli-Q water system and degassed with helium).

Preparation for the Smoking Process:

1. Mark each cigarette to the FTC insertion and butt lengths.

2. Apply #600 Scotch brand tape longitudinally on the filter of each cigarette to allow 50 % of the ventilation holes to be covered. Tape is NOT applied to the monitor cigarettes.

3. Set the smoking machine to perform two (2) clearing puffs after each run.

4. Remove the ashtray from the smoking machine and place the impingers in the smoking machine hood running the orifice of the first impinger through port #14.

PM3000376202

Source: https://www.industrydocuments.ucsf.edu/docs/mqwl0001

5. Place a Cambridge filter pad assembly into the Tygon line after the exit impinger.

6. Ensure that the puff parameters on the smoking machine are set at 45 mL, 30 seconds, and 2 seconds for puff volume, puff cycle, and puff duration, respectively. **The puff volume is measured through the impinger apparatus,** (each impinger should contain 50 mL of water during the puff volume measurement).

7. Using the Schiltknecht air flow meter, measure/adjust the airflow at port #14 of the smoking machine to 200 ± 50 mm/second. The airflow reading is taken at 40 mm from the front of the cigarette holder.

Preparation for pH analysis:

1. Check the pH electrode to make sure it is in the open position, (the fill hole must be open when taking measurements). Check the electrolyte level in the reference cavity. The level should be approximately ¼ inch below the cap. If the electrolyte level is too low, add electrolyte (4M KCl saturated with AgCl).

2. The pH electrode should be cleaned daily before calibrating the pH meter. 3% hydrogen peroxide solution is used for cleaning the pH electrode. To clean the electrode, place it in the 3% hydrogen peroxide solution for approximately 2 minutes, then remove the electrode from the solution, and rinse with Milli-Q water. Return the electrode to the pH 7 buffer and allow it to soak in the buffer for approximately ten (10) minutes prior to calibration.

3. Calibration of the pH meter is performed daily before taking any pH measurements. The pH meter is calibrated using pH 4 and pH 10 buffers for a two point standardization. The electrode slope or efficiency must be within 95% to 105% before the pH meter can be used for sample measurements.

4. The calibration standard check should be performed using the pH 7 buffer before each sample measurement. The buffer response must be within 7.00 $\pm$ 0.10 pH units or the pH meter must be recalibrated.

   Note: Between sample and standard measurements the electrode should rest in pH 7 buffer solution. Before taking the next reading, rinse the electrode with deionized water and blot dry with a tissue (Kimwipe) to prevent sample contamination.

5. The smoke from one cigarette is collected using 2 glass impingers connected in series followed by a Cambridge filter pad. 50.0 mL $\pm$ 0.2 mL of degassed Milli-Q water is used in each impinger. The Milli-Q water is degassed overnight for use that day.

6. The pH reading is taken 15.00 minutes after the last clearing puff, (the electrode is submersed for the final 5 minutes of this time period).

Test Procedure:

1. Add 50 mL of degassed water to each impinger and assemble the apparatus.

2. Insert a Cambridge filter pad holder into the Tygon tubing.

Source: https://www.industrydocuments.ucsf.edu/docs/mqwl0001

3. Prepare a pH blank by drawing nine (9) puffs on an unlit monitor cigarette through the water-containing impinger apparatus. Determine the pH of the resultant water and Cambridge filter pad. The blank is analyzed at the beginning of the testing session.

4. Per the following procedure, perform one replicate of the monitor(s) cigarette at the beginning and at the end of the testing session.

5. Insert the test cigarette, to the insertion line, into the impinger orifice.

6. Apply the microswitch handlebar across the nineteen unused smoking machine ports.

7. Apply string across port #14 at the butt length line, start the smoking machine, and light the cigarette using the Borgwaldt lighter.

8. After the cigarette is lit, remove the microswitch handlebars from the unused ports.

9. After the smoking machine has taken the final clearing puff, start the timer, and remove the impingers from the sampling apparatus, (Steps 11. through 14. need to be performed within ten (10) minutes).

10. Shake the traps vertically for ~20 seconds being careful not to spill any of the solution.

11. Transfer the contents of the two impingers and the Cambridge filter pad to a 150 mL beaker that contains a magnetic stir bar. (Note: Use finger cots when transferring the filter pads.)

12. Place the beaker on a magnetic stir plate and stir gently.

13. Ten (10) minutes after the final clearing puff, submerse the pH electrode into the beaker being careful that the electrode does not come into contact with the filter pad or the stir bar, (the electrode needs to be immersed far enough to cover both the glass pH sensing bulb and the reference junction to obtain accurate readings).

14. After five (5) minutes, record the pH.

15. Each impinger is used once per day. To clean the impinger, wash with an Alconox soap solution, rinse with deionized water, and dry overnight in an oven at 100 °C.

References:

1. Battelle Northwest, Document Code AC-3A3P-00, "Analysis of pH and Redox Potential in Mainstream Smoke Collected in an Aqueous Solution," June 24, 1997.

2. M Mangrum, Proposed R&D Analytical Method M-2-A, "Analysis of Mainstream Smoke for pH and Reduction-Oxidation Potential (Redox) in an Aqueous Solution," July 10, 1997.

Revision History:

Original version prepared September 18, 1997 by C. H. Callicutt and J. M. Garman.

Source: https://www.industrydocuments.ucsf.edu/docs/mqwl0001

Attachment I: Analysis Order:

1. Modify the smoking machine to incorporate the impinger apparatus.
2. Calibrate the smoking machine, (air flow and puff volume).
3. Calibrate the pH meter using pH 4 and pH 10 buffers.
4. Perform the calibration standard check with pH 7 buffer.
5. Determine the pH of the sample blank procedure (using an unlit cigarette).
6. Perform the calibration standard check with pH 7 buffer.
7. Determine the pH-in-smoke of IM #16 (and #15, if required).
8. Perform the calibration standard check with pH 7 buffer.
9. Determine the pH-in-smoke of the test sample.
10. Perform the calibration standard check with pH 7 buffer.
11. Determine the pH-in-smoke for the second, third and fourth replicate, performing the calibration check between each replicate.
12. Perform the calibration standard check with pH 7 buffer.
13. Analyze next sample.
14. After completion of daily testing, determine the pH-in-smoke for IM#16 (and IM#15, if required).
15. Perform the calibration standard check with pH 7 buffer.
16. Clean the smoking machine and the impingers.

    Note: Dispose of all chemicals in accordance with R&D policy.

PM3000376205

Source: https://www.industrydocuments.ucsf.edu/docs/mqwl0001